



RESEARCH DEPARTMENT

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# Construction of multilayer dielectric filters

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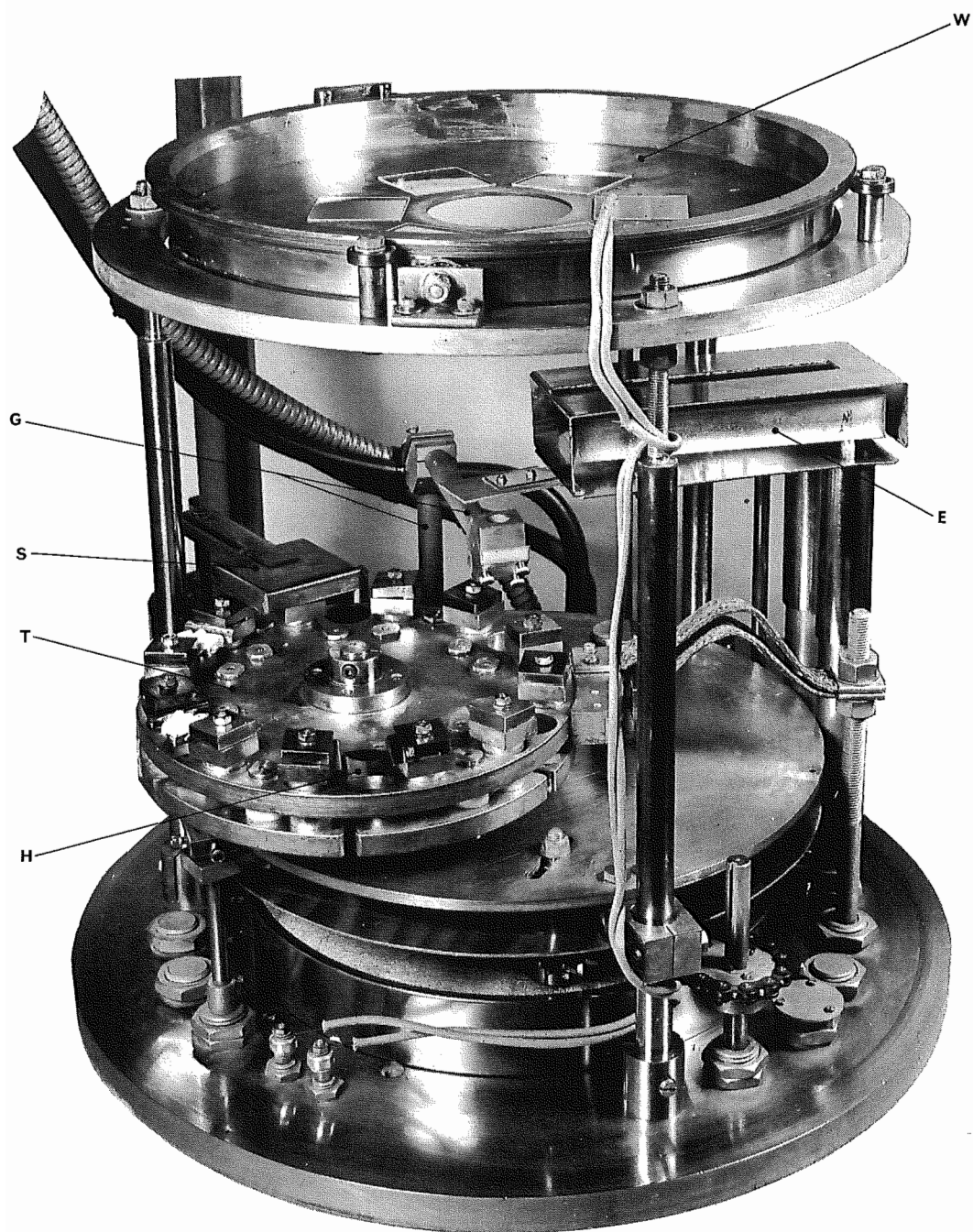
Head of Research and Development

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## CONSTRUCTION OF MULTILAYER DIELECTRIC FILTERS

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*Fig. 1 - Arrangement inside the chamber*

## CONSTRUCTION OF MULTILAYER DIELECTRIC FILTERS

### SUMMARY

*A vacuum coating plant was installed at Research Department in September 1965 and has been used for the construction of multilayer dielectric filters. Techniques developed so far for evaporating materials and monitoring the thickness of the layers are described.*

*The characteristics of some filters constructed are shown in comparison with the theoretical characteristics.*

### 1. INTRODUCTION

The principle uses of multilayer dielectric filters are

- (a) as dichroic mirrors in which incident light is split into two complementary colour components, one being reflected and the other transmitted.
- (b) as filters where a suitable absorbing type (i.e. dyed glass or gelatine) does not exist, or would be unsuitable for some other reason (e.g. in a situation where the filter would be subjected to high temperatures).

Both these types of multilayer dielectric filter are used in colour television cameras and film scanners as the colour analysis components; it is necessary that they have spectral characteristics within close tolerances and such filters are not readily available commercially. Thus it has been found useful to be able to manufacture these, especially when conducting experiments in which the analysis characteristic is the variable parameter.

The simplest type of multilayer filter consists generally of two dielectric materials of high and low refractive index with respect to glass, deposited in alternate layers on to a glass substrate; these layers are very thin (of the order of 1 micron) and are conveniently formed by evaporation of suitable materials in vacuo.

The materials used must be readily evaporable in vacuo and when allowed to condense on to the substrate must produce transparent films of sufficient homogeneity to avoid scattering of light.

Two such materials are zinc sulphide and magnesium fluoride which have high and low refractive indices respectively; using only these it has been possible to meet nearly all the filter requirements so far encountered.

### 2. EQUIPMENT

An Edwards 19E6 coating plant is used together with an Edwards MBP2A modulated-beam photometer for monitoring the film thickness. Fig. 1 shows the arrangement inside the chamber.

A turret T carries six pairs of terminals to each of which may be connected a source heater H; this may take many forms but a trough bent from tungsten foil has been found most suitable for the evaporants used. The required source is brought into the evaporating position by operation of a handwheel external to the chamber and a shutter S, operated by an external knob, is provided to terminate deposition when required. The substrate is held in the workholder W which is rotated by a variable-speed electric motor mounted below the chamber baseplate.

When evaporating, the source is positioned vertically below the periphery of the workholder; thus when the workholder is rotating, the source can be considered as a ring source below a stationary workholder. This arrangement can be shown to give very uniform deposits over a large area when the distance from the plane of the ring to the plane of the work is approximately equal to the radius of the ring; experiment shows that a slight increase in this distance is necessary to give the best uniformity over a large central area.

Two pure aluminium electrodes E, are connected to a high-voltage transformer to provide a discharge for glass cleaning; this is dealt with in Section 3.2.

Photometer beam guide tubes G direct the light from a collimated source below the edge of the baseplate, through the centre of the workholder for monitoring purposes as described in Section 3.4.

### 3. EXPERIMENTAL PROCEDURE

#### 3.1. Preparation of Evaporant

When most materials are heated in vacuo, outgassing occurs which gives rise to an increase in pressure in the chamber. For good-quality films to be produced it is necessary that the pressure be as low as possible (of the order of  $1.33 \text{ mN/m}^2$  ( $10^{-5}$  torr)) and for this reason deposition cannot be commenced until outgassing has ceased. A material such as magnesium fluoride has a relatively short outgassing time but, for zinc sulphide as supplied, it was found that outgassing caused the powder to be ejected from the heater and only very gradual heating of the material would prevent this; attempts were made to evaporate from a crucible fitted with a 'pepper pot' type cover in order to reduce the number of particles that escaped but the rather directional characteristics of this source gave rise to non-uniform coatings and precluded its use.

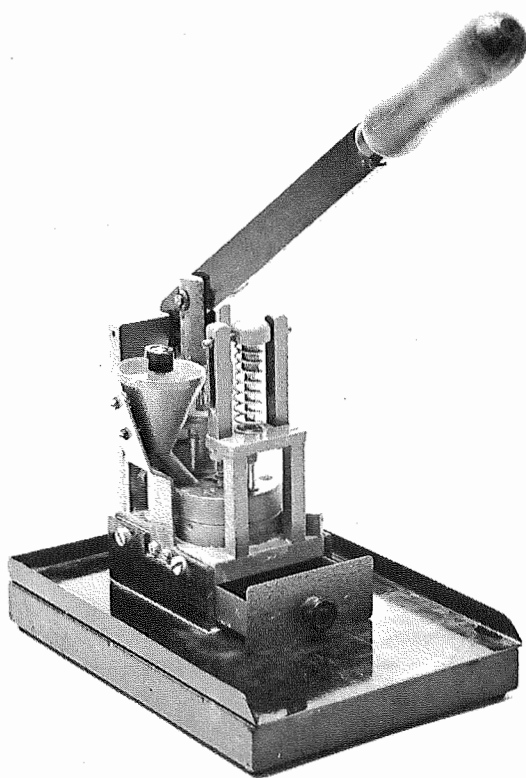


Fig. 2 - Machine for compressing powder into pellet form

Eventually it was found necessary to compress the powder into pellet form (for which purpose the small machine illustrated in Fig. 2 was constructed) and to bake the pellets in vacuo for an hour as a separate degassing process prior to evaporating.

#### 3.2. Glass Cleaning

The glass to be coated is first washed using hot water and a laboratory detergent so as to remove gross contamination; it is then dried with a clean linen cloth and any dust removed with a soft brush. Further cleaning is then carried out by supporting the glass vertically over boiling isopropyl alcohol until condensation ceases, whereupon it is ready to be fitted to the workholder and placed in the vacuum chamber.

Any remaining contamination is removed by high tension glow discharge cleaning; a high voltage (about 2 kV) is applied to two aluminium electrodes previously mentioned and the pressure in the chamber adjusted, by means of a controlled leak and reduced pumping rate, to cause a glow discharge to fill the chamber. The glass is thus bombarded with ions which clean the surface and also produce localized heating, which causes any absorbed gasses to be released; the mechanism of this cleaning process is described by Holland<sup>1</sup>.

#### 3.3. Rate of Evaporation

The most satisfactory rate of evaporation varies with the material; too high a rate may give rise to a granular structure with increased light absorption whereas a slow rate often produces soft films. Experience has shown that the best rate of deposition, in terms of layer thickness, for zinc sulphide is between 15 nm and 25 nm per min, and for magnesium fluoride between 100 nm and 200 nm per minute. The evaporation rate is controlled by the current flowing in the source heater and the quantity of material it contains.

#### 3.4. Monitoring

Fig. 3 illustrates the arrangement of the monitoring system. Lamp T, in combination with the lenses  $L_1$  and  $L_2$  and slit S, produce a parallel light beam which passes through a motor-driven chopper disc C and semi-reflecting mirror  $M_1$ . It then passes into the vacuum chamber through glass plug  $G_1$ , and is directed by means of surface reflecting mirrors  $M_2$  and  $M_3$  to the centre of the chamber and on to monitoring plate\* P. If monitoring trans-

\* In order to monitor the transmission or reflection of the layers as they are deposited, a glass plate is placed in the same plane as the glasses to be coated. This plate is at the centre of rotation of the workholder and may take the form of a small glass disc, or may be the work itself, if this is a large plate occupying most of the area of the workholder.



mission, the light leaves the chamber via glass plug  $G_2$ , and, after passing through filter  $F$ , falls upon photo-cell  $D$ . If monitoring by reflection from the plate  $P$ , the system is carefully aligned so that the beam returns along the same path to mirror  $M_1$ , where half is reflected into the photo-cell  $D'$ .

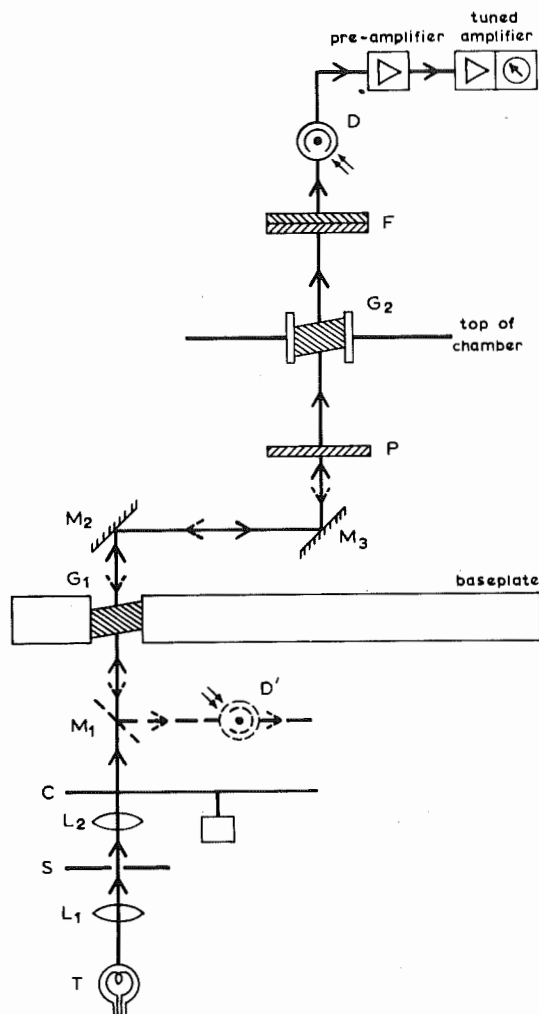


Fig. 3 - Schematic diagram of monitoring system

The output from the photo-cell is first amplified by a pre-amplifier placed close to the photo-cell housing and then by a tuned amplifier (tuned to the frequency of the chopper  $C$ ) before being fed to a suitable meter. As well as variable amplifier gain, provision is made for suppressing the meter zero thus enabling small changes in large readings to be observed with a high degree of accuracy.

Filter  $F$  is a narrow band-pass filter, the centre wavelength of which normally corresponds to the design wavelength of the dielectric filter being made, although invariably a somewhat different wavelength has to be chosen to allow for ageing effects which occur after the filter has been made and removed from the vacuum. The need for a large range of filters was overcome by means of a

narrow-band annular spectrum-wedge which was calibrated and mounted in a suitable holder so that any wavelength between 400 nm and 700 nm could be selected.

### 3.4.1. Use of the Monitoring System

In practice it has been found easiest to monitor by transmission rather than reflection because it is then not essential that the plane of the monitoring plate be precisely orthogonal to the optical axis.

At the beginning of an evaporation, the reading on the photometer meter is adjusted to read some large value, because the transmission of the filters will decrease as the number of layers, and hence the reflectance, increases.

At constant deposition rate the transmission, to narrow-band illumination, of a layer of dielectric material varies sinusoidally with time. If the material has a higher refractive index than the substrate, then the minima in transmission occur when the optical thickness of the layer is equal to an odd integer of quarter wavelengths of the monitoring light; the maxima occur at half wavelength intervals and the transmission is then substantially equal to that of the substrate. For a material of lower refractive index than the substrate, *maxima* in transmission correspond to odd multiples of a quarter wavelength.

Most multilayer dielectric filters consist of a stack of quarter wavelengths (or odd multiples thereof) of alternate high- and low-refractive index materials and, therefore, a convenient method of monitoring these is by determining the points of maximum or minimum transmission. At these maxima or minima the rate of change of transmission is zero and hence the precise quarter-wave point is difficult to determine.

A method was devised in which the transmission characteristic of a filter, layer by layer, was computed together with the effect of small changes of thickness ( $\pm 5\%$ ) on these layers. A wavelength was then chosen where the change of transmission with thickness was greatest and the filter made by monitoring to the computed transmission for each layer. The requirements for the above method to work successfully are:

- (i) a truly monochromatic monitoring light source,
- (ii) an accurate knowledge of the refractive index of the materials used, and
- (iii) very good long-term stability of the photometer.

Many filters were made using this technique, but it was eventually found more convenient to

monitor by maxima and minima, except in the case of certain special filters in which the layer thicknesses were not all simply related to the same wavelength.

As the number of layers on the monitoring plate increases, the change of transmission with layer thickness progressively decreases. It has been found that up to about ten layers can be monitored satisfactorily on one monitoring plate and this number is sufficient for most purposes. However, in order to be able to manufacture filters having a greater number of layers, a device was constructed which enabled up to six fresh monitoring plates to be used during one coating.

#### 4. AGEING EFFECTS

After removal from the vacuum chamber the filter undergoes a change of characteristic due to a change in refractive index of one or both of the materials on exposure to air and atmospheric pressure. This 'ageing effect' is complete after about three hours and no further change appears to take place. Heating the filter does not accelerate this process but actually halts it; on cooling the ageing continues.

#### 5. DURABILITY

Zinc sulphide has poor resistance to abrasion and, in combination with layers of magnesium fluoride, may sometimes 'peel off' when breath condenses upon its surface; a worthwhile improvement can be made by heating the filter in an oven at 150°C for about an hour and allowing it to cool slowly.

Magnesium fluoride is very resistant to scratching and moisture, especially if deposited on to glass pre-heated to 200°C (a radiant heater is built into the top of the chamber to enable this to be done); these properties together with its low refractive index make it the most suitable material for anti-reflection coating ('bloom') and it has been used as such very successfully.

#### 6. ALTERNATIVE MATERIALS

A search has been made for a more durable high-refractive index material to replace zinc sulphide; possible alternatives are cerium dioxide, zirconium dioxide, silicon monoxide and titanium dioxide, which all require a very high temperature for evaporation.

Cerium dioxide has been evaporated from a tungsten heater and the coating produced was very hard; the refractive index was found to increase with the temperature of the substrate and at the maximum temperature attainable with the existing plant (300°C) a refractive index of 2.0 was achieved.

However, in order to make highly reflecting filters with a reasonable number of layers it is necessary that the refractive index of the high-index layers shall be in excess of 2.2; thus the material is unsuitable when evaporated under these conditions. Furthermore, it was found that the tungsten heater was attacked by cerium dioxide.

It was found impossible to evaporate zirconium dioxide, a temperature greater than the melting point of tungsten being required.

Silicon monoxide was not examined because the value of refractive index quoted in the literature was only 1.9 and furthermore varied according to the rate of evaporation.

A satisfactory method of producing films of titanium dioxide is to first deposit a layer of pure titanium in vacuo and then heat this in air to form the dioxide. Alternatively the titanium may be sputtered\* in air, but this is very slow; in addition, the required equipment to do this is not included with the existing plant. Many successful films have been made by heat oxidation of pure titanium but this technique is limited to one layer only and is therefore of no use for multilayer filters.

During the last few years a technique has been developed elsewhere for heating materials in vacuo by electron bombardment; by this method temperatures up to about 4000°C may be attained and thus materials such as zirconium dioxide may be more easily evaporated. The material to be evaporated is contained in a conducting crucible (e.g. carbon) and, since the material itself, rather than the container, is heated by the electrons, it does not easily react with the container.

#### 7. CHARACTERISTICS OF SOME FILTERS CONSTRUCTED

Figs. 4, 5, 6 and 7 show a comparison of the practical and theoretical characteristics of some filters constructed. Fig. 4 shows the spectral transmission characteristic of a seven-layer filter in which each layer has an optical thickness of one quarter of the design wavelength; this is a first order interference filter. A useful modification to this filter is shown in Fig. 5, in which a low-refractive index layer having an optical thickness of one half of the design wavelength has been added<sup>2</sup>. This results in the reduction of the amplitude of the 'bump' in the characteristic of the filter of Fig. 4 occurring at 510 nm.

\* Sputtering is a technique of deposition in which an electrical discharge is passed between two electrodes at low pressure; the cathode is formed from the material to be deposited and, under ionic bombardment, slowly disintegrates. The liberated atoms may react with the residual atmosphere and then condense on surrounding surfaces.

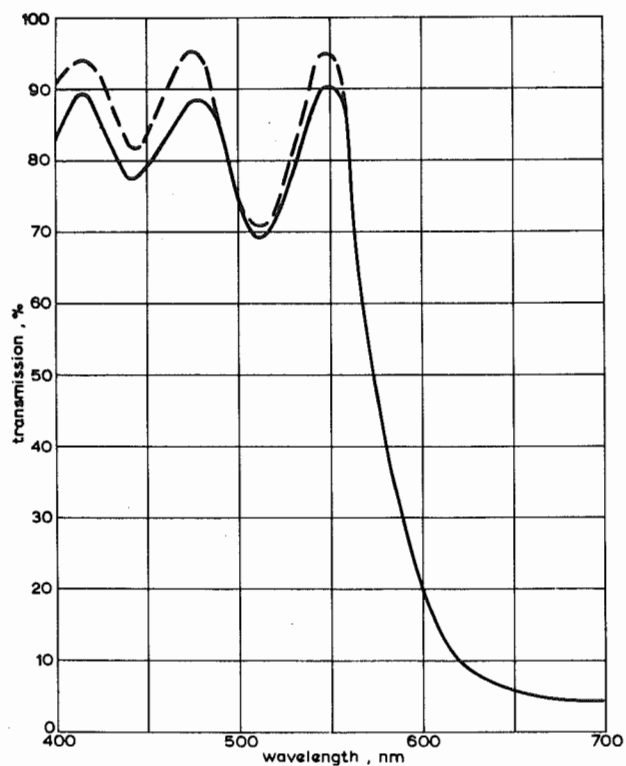


Fig. 4 - Spectral transmission characteristic of seven-layer first order filter

7 layers  $\lambda/4$  ZnS +  $\lambda/4$  MgF<sub>2</sub>.

$\lambda = 700$  nm

—— practical      - - - - - theoretical

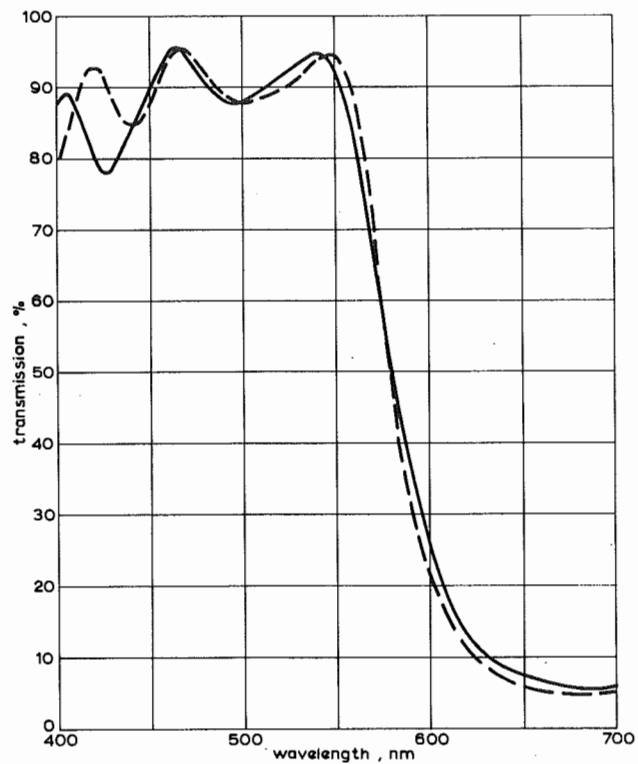


Fig. 5 - Spectral transmission characteristic of eight-layer first order filter

7 layers  $\lambda/4$  ZnS +  $\lambda/4$  MgF<sub>2</sub> + 1 layer  $\lambda/2$  MgF<sub>2</sub>.

$\lambda = 700$  nm

—— practical      - - - - - theoretical

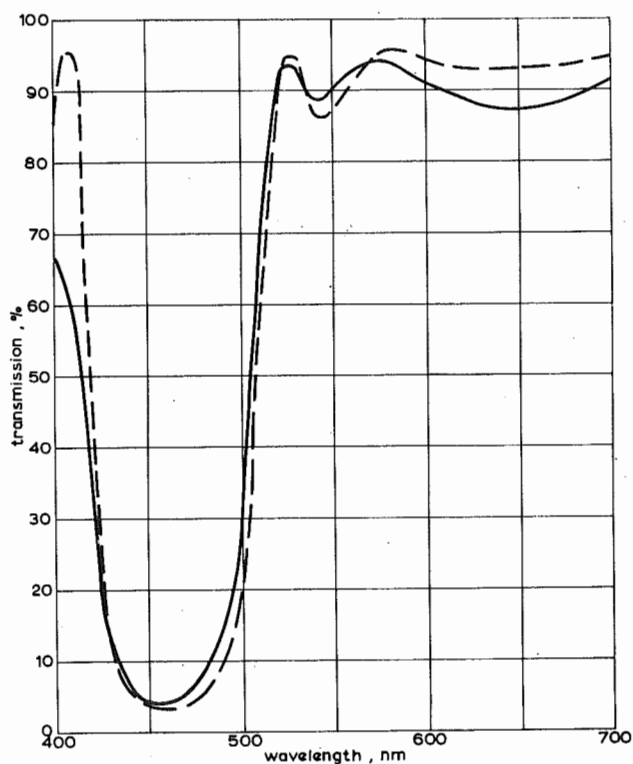


Fig. 6 - Spectral transmission characteristic of eight-layer, second order high-index filter

7 layers  $3\lambda/4$  ZnS +  $\lambda/4$  MgF<sub>2</sub> + 1 layer  $\lambda/2$  MgF<sub>2</sub>.

$\lambda = 460$  nm

—— practical      - - - - - theoretical

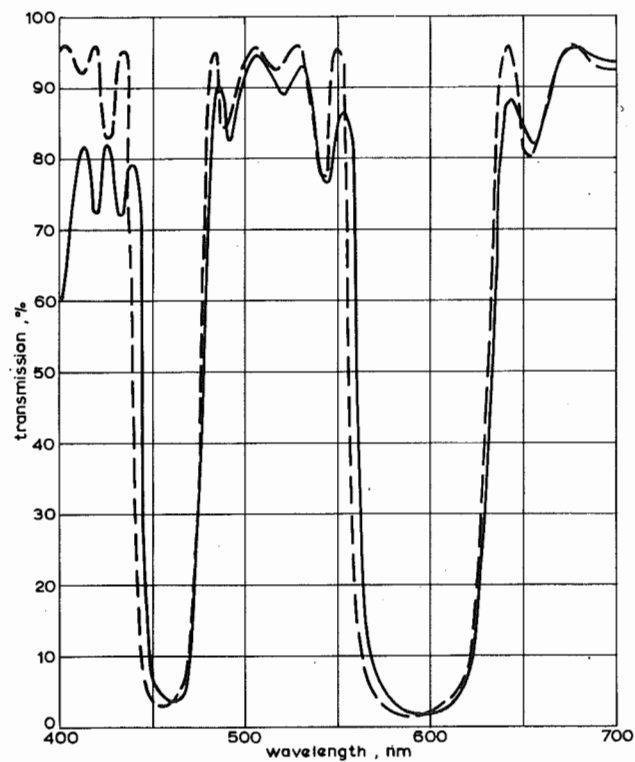


Fig. 7 - Spectral transmission characteristic of ten-layer, third order high-index filter

9 layers  $5\lambda/4$  ZnS +  $\lambda/4$  MgF<sub>2</sub> + 1 layer  $\lambda/2$  MgF<sub>2</sub>.

$\lambda = 600$  nm

—— practical      - - - - - theoretical

The addition of this last layer has been adopted for most filters constructed; the filters shown in Figs. 6 and 7 are of this type.

The transmission characteristics shown in Figs. 6 and 7 are those of filters in which the thickness of the high-refractive index material has been increased. In Fig. 6 the optical thickness is three quarters of the design wavelength and in Fig. 7 five quarters, (i.e. second and third order respectively).

The close agreement between theoretical and practical characteristics shown here can only be obtained when the correct monitoring wavelength has been chosen and sufficient care is taken in determining the points of maximum and minimum transmission whilst monitoring. It will be observed in Figs. 6 and 7 that the agreement between the practical filter and the theoretically computed one is less close in the short-wavelength region (near to 400 nm). This lack of agreement is believed to be due to absorption of light by the zinc sulphide.

## 8. CONCLUSIONS

Techniques have been established for the production of multilayer dielectric filters using magnesium fluoride and zinc sulphide. The inadequate durability of zinc sulphide as a high-refractive index material has led to the investigation of other materials and the conclusion that further work using electron-bombardment techniques is necessary.

## 9. REFERENCES

1. HOLLAND, L. 1956. Vacuum deposition of thin films. London, Chapman and Hall, 1956, pp. 74 – 80.
2. The construction and design of dichroic mirrors. BBC Research Department Report No. T-066, Serial No. 1957/13.